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ANALYTICAL REFERENCE SERVICE TRAINING PROGRAM. AIR LEAD, NUMBER 1, NOVEMBER 1961

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio

November 1961

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ANALYTICAL REFERENCE SERVICE
AIR-LEAD NUMBER 1

Prepared by the
Analytical Reference Service

TRAINING PROGRAM

U.S. DEPARTMENT OF HEALTH, EDUCATION AND WELFARE
Public Health Service
Bureau of State Services

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio

1961

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ACKNOWLEDGEMENT

The Analytical Reference Service Staff gratefully acknowledges the assistance of A. W. Briedenbach, Ph. D., Assistant Chief, Air Pollution Training Activities.

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FOREWORD

The Analytical Reference Service is conducted by the Robert A. Taft Sanitary Engineering Center to provide a means of evaluating laboratory methods in the field of environmental sciences and engineering. Membership includes federal, state and municipal agencies, industries, universities, consulting firms, and foreign agencies. Cooperative studies by member organizations, through analysis of identical samples of environmental media and critical review of methodology, provide the mechanism for:

1. Statistical evaluation of procedures, including precision and accuracy.

2. Comparison of analytical procedures and results between laboratories having similar responsibilities.

3. Exchange of information regarding weaknesses of currently approved methods.

4. Further development of methods or entirely new approaches.

5. Broad-scale evaluation of new analytical methods.

Initially directed toward examination of water, the studies have been broadened to include other media. Samples are designed and prepared to contain measured amounts of selected constituents. Decisions as to qualitative makeup are made by the staff of ARS, the membership, and consultants. Notice of a specific study is forwarded to each member with information regarding the qualitative makeup of the sample and special details regarding its handling.

Following indication of a member's desire to participate in the specific study, a portion of the sample is shipped with accompanying data forms which provide space for numerical analytical values and a narrative critique of the procedures used. The laboratories are urged to comment on the methods, difficulties, modifications, sources of error, and other factors. Results are requested within sixty days.

The results of each study are compiled, as in the accompanying report. Experience indicates that a repeat study produces a sizeable increase in the number of participants. The first study designed to evaluate analytical methods for air pollutants was completed in 1958. Twenty-one agencies participated in the 1958 study. Forty-one agencies cooperated in this study.

A water-metals study has recently been completed, and a report is being prepared. A water-trace elements sample, containing beryllium, boron, arsenic, and selenium, was shipped to members in October. A milk sample is planned for the determination of DDT by a specified method. Studies for the determination of strontium-90 in water and SO_2 in air are in the planning stage.

Selection of these studies was guided by answers to the questionnaires distributed to member agencies to poll their interests. Results of this poll will continue to serve as a basis for planning future studies.

Membership now consists of 120 agencies of many diversified interests. The steadily increasing membership strongly indicates the broad interest in and need for this type of service.

H. P. Kramer
Chief, Training Program

ANALYTICAL REFERENCE SERVICE MEMBERS*

STATE AGENCIES

Alabama Water Improvement Commission
Arizona State Department of Health
Arkansas State Board of Health
California State Department of Public Health (Berkeley)
California State Department of Public Health (Los Angeles)
California Department of Water Resources
California Disaster Office
Colorado State Department of Public Health
Connecticut State Department of Health
Delaware Water Pollution Commission
Florida State Board of Health (Bureau of Laboratories, Jacksonville)
Florida State Board of Health (Sanitary Engineering, Jacksonville)
Florida State Board of Health (Pensacola)
Florida State Board of Health (Winter Haven)
State of Hawaii Department of Health
Idaho Department of Health
Illinois State Department of Public Health (Chicago)
Illinois State Department of Public Health (Springfield)
Illinois State Water Survey Division
Indiana State Board of Health
Kansas State Board of Health (Sanitary Engineering Laboratories)
Kentucky State Department of Health
Louisiana State Board of Health
Maryland State Department of Health
Maryland State Water Pollution Control Commission
Massachusetts Department of Public Health
Massachusetts General Hospital
Michigan State Department of Health
Michigan Water Resources Commission
Minnesota State Department of Health
Missouri Department of Public Health & Welfare
Montana State Board of Health
Nebraska State Department of Health
Nevada State Department of Health (Las Vegas)
Nevada State Department of Health (Reno)
New Hampshire State Department of Health
New Jersey Department of Health
New Mexico State Department of Public Health

*November, 1961

New York State Department of Health
New York State Conservation Department
North Carolina Department of Water Resources
Ohio State Department of Health
Oklahoma State Department of Health
Oregon State Board of Health
Pennsylvania Department of Health
Rhode Island Department of Health
South Carolina Water Pollution Control Authority
South Dakota Department of Health
Tennessee Stream Pollution Control Board
Texas State Department of Health
Utah State Department of Health
Vermont State Department of Health
Vermont State Department of Water Resources
Virginia State Department of Health
Virginia State Water Control Board
Washington State Department of Health
West Virginia State Water Resources Commission
Wisconsin State Board of Health

MUNICIPAL AGENCIES

Metropolitan Water District of Southern California
Los Angeles Department of Water and Power
Los Angeles County Flood Control District
Los Angeles Department of Public Works (Hyperion Treatment Plant)
City of Pasadena
Chicago Department of Water and Sewers
Topeka, Kansas Water Works
Flint Water Plant, Department of Public Works & Utilities
St. Louis County Water Company
St. Louis Department of Health and Hospitals
Metropolitan Utilities District (Omaha, Nebraska)
City of Durham Department of Water Resources
Dayton Department of Service and Buildings
Philadelphia Suburban Water Company
Philadelphia Water Department (Torresdale Laboratory)
Philadelphia Water Department (Belmont Laboratory)
Philadelphia Department of Public Health

FEDERAL AGENCIES

Food and Drug Administration, Division of Pharmacology, Washington, D.C.
Tennessee Valley Authority, Wilson Dam, Alabama (Occupational Health Branch)
Tennessee Valley Authority, Chattanooga, Tennessee (Stream Pollution Control)
2794th U. S. Air Force Dispensary - Class B
U. S. Army Environmental Hygiene Agency, Maryland
U. S. Dept. of the Interior, Bureau of Reclamation, Denver, Colorado
U. S. Dept. of the Interior, Geological Survey, Sacramento, California
U. S. Dept. of the Interior, Geological Survey, Denver, Colorado
U. S. Dept. of the Interior, Geological Survey, Columbus, Ohio
U. S. Dept. of the Interior, Geological Survey, Philadelphia, Pennsylvania
Pearl Harbor Naval Shipyard
DHEW, PHS Great Lakes - Illinois River Basins Project
DHEW, PHS Off-Site Radiological Safety Program, Las Vegas, Nevada
Associated Universities, Inc.

UNIVERSITIES

Case Institute of Technology, Dept. of Civil and Sanitary Engineering
Columbia University
Georgia Institute of Technology, Dept. of Applied Biology
University of Pittsburgh, Graduate School of Public Health
Purdue University, Department of Chemistry
Rutgers - The State University (Department of Sanitation)
University of Kansas, School of Engineering and Architecture
Johns Hopkins University, School of Hygiene and Public Health
University of Florida, Department of Chemistry

INDUSTRY

American Cyanamid Company, Bound Brook, New Jersey
Atlantic Refining Company, Philadelphia, Pennsylvania
Battelle Memorial Institute, Columbus, Ohio
California Water Service Company, San Jose, California
Central Farmers Fertilizer Company, Montpelier, Idaho

Culligan, Inc., Northbrook, Illinois
Dearborn Chemical Company, Chicago, Illinois
E.I. du Pont de Nemours & Co., Aiken, South Carolina
Ekroth Laboratories, Inc., Brooklyn, New York
Food Machinery & Chemical Corp., Pocatello, Idaho
Goodyear Atomic Corporation, Portsmouth, Ohio
Hagan Chemicals & Controls, Inc., Pittsburgh, Pennsylvania
Industrial Chemicals, Inc., South Bend, Indiana
Ionics, Incorporated, Cambridge, Massachusetts
Kennecott Copper Corporation, Salt Lake City, Utah
Monsanto Chemical Company, St. Louis, Missouri
NALCO Chemical Company, Chicago, Illinois
Radiation Detection Company, Palo Alto, California
Reynolds Electrical & Engineering Co., Las Vegas, Nevada
Roy F. Weston, Inc., Newtown Square, Pennsylvania
Sandia Corporation, Sandia Base, Albuquerque, New Mexico
Tracerlab, Inc., Richmond, California
Water Service Laboratories, Inc., New York, New York

FOREIGN

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Ottawa, Ontario, Canada, Department of National Health & Welfare
Ottawa, Ontario, Canada, Occupational Health Division, Department of National Health & Welfare
Caracas, Venezuela, Instituto Nacional de Obras Sanitarias
Kingston, Jamaica, West Indies, Scientific Research Council

PARTICIPANTS IN THIS STUDY

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Connecticut State Department of Health
Florida State Board of Health (Bureau of Laboratories, Jacksonville)
Idaho Department of Health
Illinois State Department of Public Health (Chicago)
Louisiana State Board of Health
Maryland State Department of Health
Michigan State Department of Health
Minnesota State Department of Health
Missouri Department of Public Health & Welfare
Montana State Board of Health
New York State Department of Health
Rhode Island Department of Health
Utah State Department of Health
Vermont State Department of Health
Vermont State Department of Water Resources
Washington State Department of Health
Wisconsin State Board of Health
Los Angeles Department of Water and Power
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GUEST PARTICIPANTS

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Bay Area Air Pollution Control District
California Department of Public Health, Berkeley (Air & Industrial Hygiene)
Fresno County Public Health Department (Division of Laboratories)
Georgia Department of Public Health (Industrial Hygiene Section)
Hawaii Department of Health (Air Sanitation Section)
Kentucky Department of Health (Occupational Health)
Minneapolis Health Department

Minnesota, University of
New Hampshire State Health Department (Occupational Health)
New Jersey Department of Health (Occupational Health)
New York Department of Air Pollution Control
Ohio State Department of Health (Industrial Hygiene)
Orange County Air Pollution District, Anaheim, California
Pennsylvania Department of Health (Air Pollution Control Section)
Philadelphia Department of Health (Air Pollution Control Laboratory)
Tennessee Department of Public Health (Industrial Hygiene Laboratory)
Virginia State Department of Health (Industrial Hygiene Laboratory)

STUDIES ON WHICH REPORTS HAVE BEEN COMPLETED

Water-Minerals	Calcium, magnesium, hardness, sulfate, chloride, alkalinity, nitrite, nitrate, sodium and potassium. Studies were completed in 1956, 1958 and 1961.
Water-Metals	Lead, copper, cadmium, aluminum, chromium, iron, manganese, and zinc. A study was completed in 1957 and a repeat study is in the report stage and will be completed in 1961.
Water-Fluoride	Fluoride in the presence and absence of interferences with and without distillation, using a specified procedure. Studies were completed in 1958 and 1961.
Water-Radiological	Gross beta and gamma activity. Studies were completed in 1959 and 1961.
Water-Surfactant	Surfactant in various waters. A study was completed in 1959.
Water-Oxygen Demand	Biochemical oxygen demand and chemical oxygen demand. A study was completed in 1960.
Air-Inorganics	Chloride, sulfate, fluoride and nitrate in aqueous solution and on glass fiber hi-vol filter mats. A study was completed in 1958.

STUDIES IN PROGRESS

Water-Metals	Copper, cadmium, aluminum, chromium, iron, manganese, zinc and lead. Report is being prepared.
Water-Trace Elements	Arsenic, boron, selenium and beryllium.

AIR-LEAD NUMBER 1

PURPOSE OF STUDY

In the field of air pollution, awareness of the pollution potential of vehicular exhaust has resulted in studies aimed at measuring levels of exhaust components. Since excessive exposure to lead has long been recognized as a serious public health problem, a significant portion of this work is directed currently toward the measurement of lead concentrations in urban atmospheres. A variety of chemical procedures are available for the determination of lead. Most of them represent modifications of procedures originally developed for the determination of this contaminant in media other than air or in concentrations higher than those usually present in the atmosphere.

Since reliable analytical methods are the indispensable tool of any study of this nature, a need was recognized for an evaluation of methods now in use in terms of accuracy and precision. This study was therefore undertaken to provide such comparative information to those who have responsibilities or interest in the measurement of lead in the atmosphere.

PREPARATION OF SAMPLE

The air-lead samples were prepared on filter-paper tape to simulate, as far as possible, the appearance of filters through which a cubic meter of air had been passed. The amount of lead added to each spot was in agreement with the amount found in samples above street level during moderate traffic conditions. No interfering substances were added. Activated carbon was used to simulate soot and other soiling materials usually deposited on filters during a sampling period.

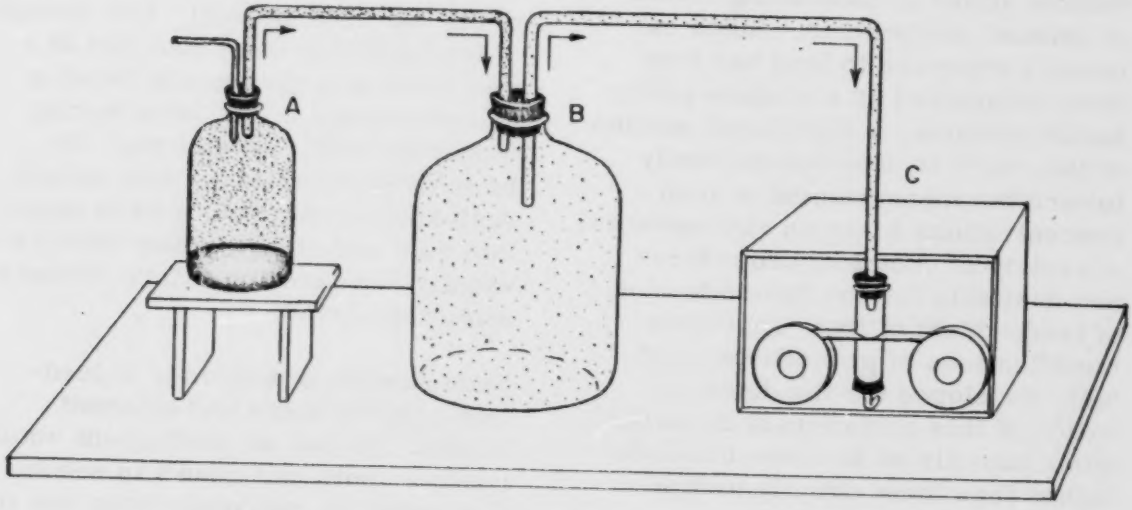
Each sample consisted of 5 lead-impregnated spots and adjacent blanks. So that no participant would receive spots and blanks in sequence, they were cut randomly from one roll of tape. The 5 spots and the 5 blanks were placed in separate envelopes. Results from any 3 of the spots and blanks were requested.

An apparatus consisting of 3 chambers was used (Figure 1). Finely divided, de-leaded activated charcoal was placed in the primary chamber A. When the pump in the Hemeon Sampler, C, was operating, the unsettled particles of carbon in chamber A passed

through chamber B into the sampler where they were deposited on Whatman #4 filter tape. Use of chamber B permitted a longer sampling period and produced spots of uniform density. The spots were spaced to permit sufficient adjacent tape to serve as blanks.

One drop of standard solution containing 5.0 micrograms of lead was added to the center of each spot from a microburet fitted with a hypodermic needle for a delivery tip.

Figure 1. SAMPLE PREPARATION APPARATUS



DISCUSSION OF ANALYTICAL METHODS

EXTRACTION PROCEDURE

Since no procedure for extracting the lead was specified, a number of methods were reported. For the purpose of evaluation, the methods were divided as shown in Table 1.

Table 1. DIVISION OF METHODS

Method of Extraction	Users
Wet ash*, HNO_3 only	13
Digestion**, HNO_3 + H_2SO_4 + $HClO_4$	7
Digestion, mixture of two acids	6
Pulped and filtered, HNO_3 only	9
Other	3
Unspecified	4
	42

*"Wet ash" is defined as the addition of acid to the sample to effect decomposition of the organic matter by heating to dryness.

**"Digestion" is defined as the addition of acid or acids to the sample to effect decomposition of the organic matter without heating to dryness.

ANALYTICAL PROCEDURES

Of the 42 participating laboratories, 3 used the spectrograph for analyzing the extracted lead; 27 used the mixed-color dithizone procedure; 11 used the monocolor dithizone procedure; 1 participant used a green monocolor dithizone method.

Dithizone Methods

Two basic dithizone methods were used, monocolor and mixed color. In both methods, dithizone in an organic solvent reacts with lead to form a complex salt called a dithizonate. Excess dithizone is necessary to insure complete reaction.

In mixed-color methods, the excess dithizone and varying amounts of lead dithizonate produce colors in the organic solvent layer ranging from green through blue to red. These colors are read either visually or photometrically.

In monocolor methods, the excess dithizone reagent is removed from the organic solvent leaving only the red-colored lead dithizonate to be read photometrically.

In the green monocolor method used by one participant, the complexed dithizone was reconverted to free

dithizone and read photometrically at the appropriate wavelength.

The two basic dithizone methods can be divided further into single or double extraction methods. In double extraction methods an additional chemical operation is involved. Lead is removed from interfering metals by lowering the pH which converts lead dithizonate to a compound soluble in the aqueous phase. After separation, the pH of the aqueous lead solution is again adjusted for subsequent organic extraction and photometric measurement.

Spectrographic Methods

Two of the 3 participants who used the spectrograph did not specify their methods. The third used indium as the internal standard.

The polarograph was not used by any participant in this study.

TREATMENT OF DATA

INDIVIDUAL RESULTS

Most participants reported their corrected* triplicate results to tenths of a microgram. Results which were carried beyond tenths of a microgram were rounded off as specified in Standard Methods for the Examination of Water and Wastewater (11th Edition, 1960, page 18). The average of each set of triplicate results (Column A, Table 3) was calculated and also rounded off to a tenth of a microgram (Column C). Some participants reported all 5 results, using several comparative methods. In these instances, the 3 results obtained by any 1 method were selected as the report from that participant.

Precision of analysis within the sets of triplicate results (Column A) was assessed by calculating the average percent deviation. This percentage was obtained by averaging the deviations from the mean of each of the three reported values (Column B). This average was then divided by the mean, and the resulting quotient was expressed as percent deviation (Column C).

Accuracy of the sets of triplicate results is represented as percent deviation from the amount of lead added (Column D). This percent deviation was arrived at by averaging the deviations of the individual results from the amount added (5.0 µg). This average was then divided by the amount added, and

*Corrected individual result = (individual result) - (average of blank results)

the resulting quotient was expressed as percent deviation.

TOTAL RESULTS

The mean, median, and standard deviation were calculated. The true mean was 5.02 μg. When rounded off to 5.0, it was equal to the amount of lead added and was used to compute the standard deviation. The summary of computations is listed in Table 2.

Table 2.
COMPUTATIONS FROM THE DATA

Computed value	Micrograms
Amount of lead added	5.00
Mean	5.02
Median	5.15
Standard deviation	±1.71

Table 3. ANALYTICAL DATA (Amount of lead added = 5.0 µgs)

Lab. no.	A µgs lead reported	B Mean	C Av. % deviation within the lab.	D % deviation (amt. added)
41	1.4 1.4 1.7	1.5	8.7	70.0
4	2.0 2.3 2.1	2.1	4.8	57.4
26	1.8 2.4 2.6	2.3	13.0	54.6
12	3.1 3.4 4.2	3.6	11.9	28.6
14	3.7 4.1 3.1	3.6	10.3	27.4
37	4.3 3.3 3.3	3.6	11.9	27.4
25	4.9 2.4 4.4	3.9	25.6	22.0
35	4.6 3.8 3.6	4.0	10.0	20.0
11	5.2 4.8 3.2	4.4	18.4	14.6
39	4.2 4.6 4.4	4.4	4.6	12.0

Lab. no.	A µgs lead reported	B Mean	C Av. % deviation within the lab.	D % deviation (amt. added)
40	4.4 4.4 4.4	4.4	0.0	12.0
20	4.5 4.5 4.5	4.5	0.0	10.0
2	5.0 4.4 4.4	4.6	5.8	8.0
28	4.6 4.4 4.9	4.6	3.7	7.4
5	4.7 4.7 4.7	4.7	0.0	6.0
33	4.9 5.0 4.9	4.9	0.6	1.4
10	5.0 4.9 5.1	5.0	1.4	1.4
16	4.9 5.0 5.0	5.0	0.6	0.6
8	6.0 4.3 ---	5.1	16.7	17.0
32	5.6 4.7 5.0	5.1	6.5	6.0

Lab. no.	A µgs lead reported	B Mean	C Av. % deviation within the lab.	D % deviation (amt. added)
SEC	4.9 5.3 5.2	5.1	0.2	4.0
13	5.0 5.0 5.5	5.2	4.4	3.4
15	5.3 5.0 5.4	5.2	1.9	4.6
29	5.0 5.2 5.4	5.2	2.5	4.0
30	5.2 5.1 5.4	5.2	1.9	4.6
1	4.9 3.7 7.3	5.3	25.1	24.6
18	5.4 5.6 4.8	5.3	5.7	8.0
19	5.6 5.2 5.2	5.3	13.2	6.6
31	7.0 4.7 4.2	5.3	21.3	20.6
38	6.1 4.7 5.0	5.3	10.8	9.4
22	5.5 5.2 5.5	5.4	2.4	8.0

Lab. no.	A µgs lead reported	B Mean	C Av. % deviation within the lab.	D % deviation (amt. added)
36	5.7 4.7 5.7	5.4	8.0	11.4
21	5.6 6.0 4.9	5.5	7.3	11.4
23	5.1 5.1 6.4	5.5	10.4	10.6
24	6.0 5.0 6.0	5.7	7.5	13.4
6	5.7 5.7 5.9	5.8	1.7	15.4
34	5.8 6.0 5.8	5.9	1.7	17.4
3	5.6 6.8 6.1	6.2	7.0	23.4
27	6.3 6.5 5.7	6.2	4.8	23.4
9	6.3 6.3 6.3	6.3	0.0	26.0
17	12.2 1.7 14.7	9.5	55.5	134.6
7	10.3 10.3 9.0	9.9	5.7	97.4

Figure 2. EXTRACTION METHODS FOR DITHIZONE ANALYSIS

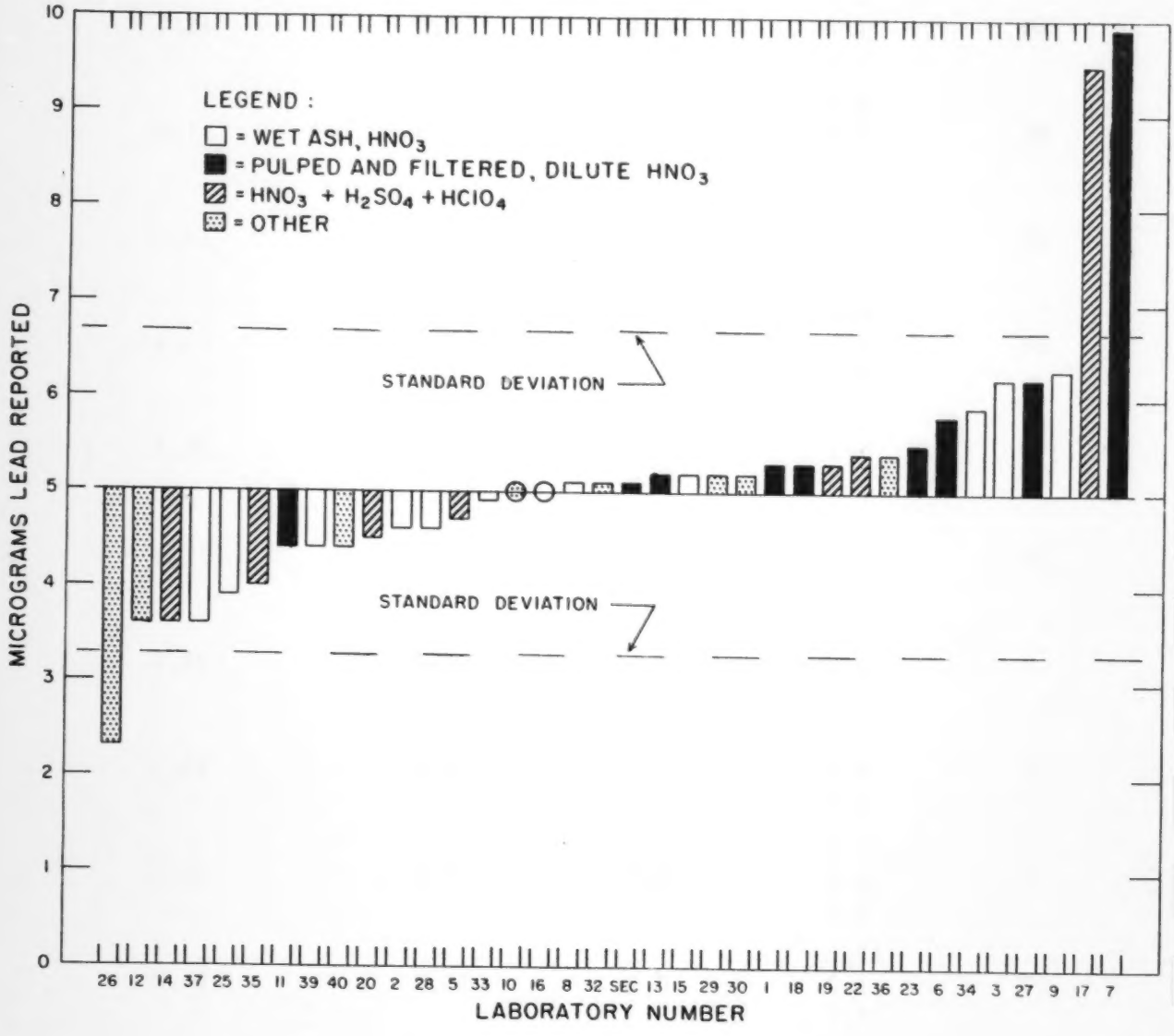
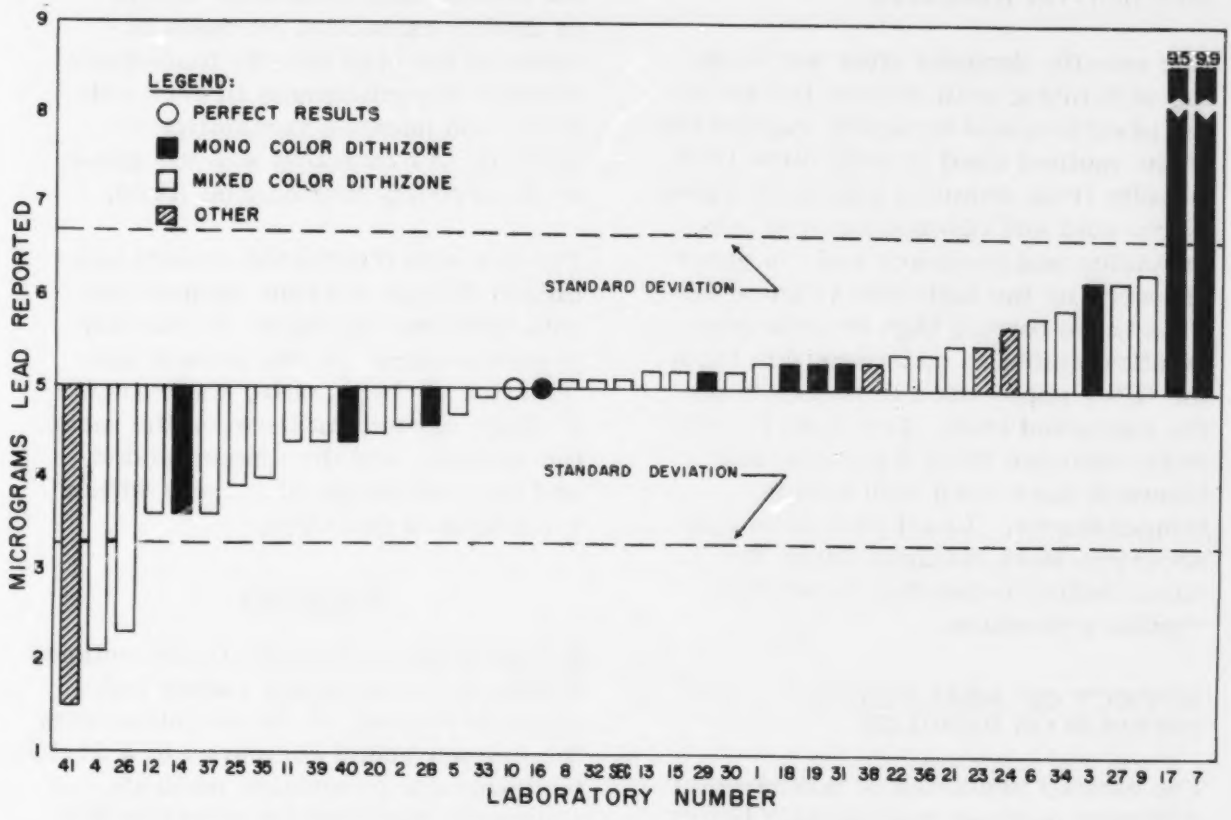

LEGEND :
□ = WET ASH, HNO_3
■ = PULPED AND FILTERED, DILUTE HNO_3
▨ = HNO_3 + H_2SO_4 + $HClO_4$
▩ = OTHER
STANDARD DEVIATION
STANDARD DEVIATION
MICROGRAMS LEAD REPORTED
0 1 2 3 4 5 6 7 8 9 10
26 12 14 37 25 35 11 39 40 20 2 28 5 33 10 16 8 32 SEC 13 15 29 30 1 18 19 22 36 23 6 34 3 27 9 17 7
LABORATORY NUMBER

Figure 3. METHODS FOR DETERMINING LEAD


LEGEND:
PERFECT RESULTS
MONO COLOR DITHIZONE
MIXED COLOR DITHIZONE
OTHER
STANDARD DEVIATION
STANDARD DEVIATION
MICROGRAMS LEAD REPORTED
9.5 9.9
9 8 7 6 5 4 3 2 1
41 4 26 12 14 37 25 35 11 39 40 20 2 28 5 33 10 16 8 32 SEC 13 15 29 30 1 18 19 31 38 22 36 21 23 24 6 34 3 27 9 17 7
LABORATORY NUMBER

DISCUSSION OF RESULTS

EFFECT OF EXTRACTION METHOD ON RESULTS

The results obtained after wet washing with nitric acid showed the greatest precision and accuracy regardless of the method used to determine lead. Results from samples pulped in dilute nitric acid and filtered showed less precision and accuracy and, in general, were on the high side (Figure 2). This trend toward high results may be attributable to contamination from the filter paper used in transferring the extracted lead. Low lead results were reported from 4 participants known to have used high ashing temperatures. Least precision and accuracy were obtained using the nitric-sulfuric-perchloric acid digestion procedure.

EFFECT OF ANALYTICAL METHOD ON RESULTS

The overall precision of mixed-color dithizone methods was slightly better than that of monocolor methods. As a group, results obtained with the monocolor methods showed twice the percent deviation from the amount added as the percent deviation obtained with mixed-color methods. Figure 3 (Methods for Determining Lead) shows that, as a group, results of monocolor dithizone methods are generally high and those of mixed-color methods are generally low.

No comparison was made between the mixed- and monocolor single or double extraction methods because of the statistically inadequate number of participants in each category, and because variability in methods of extraction was too great to observe any pronounced trend.

The few spectrographic results submitted did not warrant comparison with dithizone methods. Particularly encouraging are the normal distribution of the results, the extremely close agreement between the mean, the median, and the amount added, and the percentage of results within $\pm$ 1 standard deviation.

SUMMARY

Examination of Column-D percentages (Table 3) for accuracy values indicates an overall 16.3% deviation from the amount added compared to a 31.1% deviation for monocolor methods. Column-C numbers for precision did not indicate as great a difference: the mixed-color methods showed an overall 7.0% deviation within laboratories as compared to 11.4% for monocolor methods.

Within the conditions established for this study, the results indicate that the nitric acid, wet ashing method for extraction, together with a mixed-color dithizone procedure, is the

method of choice for determining lead on filter paper in the 0- to 10-microgram range.

Further study evaluating the effect on precision and accuracy of interfering substances present in actual air samples would be valuable.

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MONOCOLOR DITHIZONE METHODS

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*References are given only when cited by more than one participant. No methods of extraction are referenced.